भारतीय मानक
Indian Standard

IS 15131 : 2020

2, 6-डाईब्रोमो-4-नाइट्रोएनीलीन — विशिष्टि

(पहला पुनरीक्षण)

2,6 Dibromo-4-Nitroaniline — Specification

(First Revision)

ICS 71.080.80

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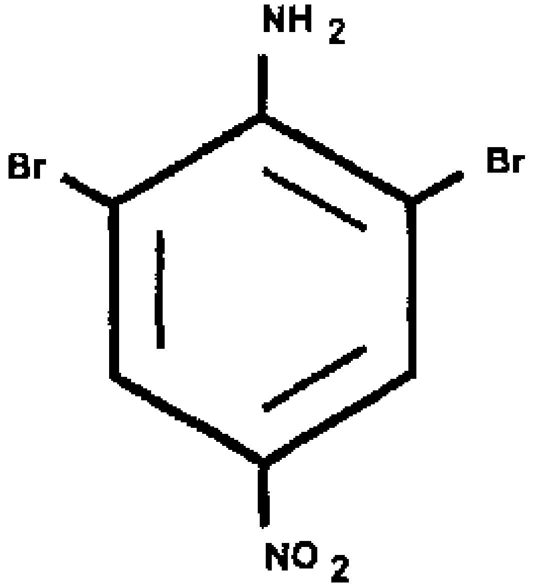
September 2020 **Price Group 6**

Dye Intermediates Sectional Committee, PCD 26

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

2,6 Dibromo-4-Nitroaniline is an important dyestuff intermediate used in the manufacture of azo disperse dyes. It has the following structural formula:



2,6 Dibromo-4-nitroaniline
Molecular mass = 296
[CAS Registry No.: 827-94-1]

This standard was first published in 2002. The Committee responsible for the formulation of this standard has decided to revise it in order to incorporate recent development in analytical techniques and use of more sophisticated instrument to determine purity and impurity profile. Test method for purity determination by High Performance Liquid Chromatography (HPLC) and also impurity of 2,4-Dichloro-4-nitroaniline content by HPLC and 4-Nitroaniline and 2-Bromo-4-Nitroaniline by GC are incorporated. In addition to these, specification for Chlorobenzenes and Chlorotoluenes are also incorporated considering to environmental hazard of these derivatives.

The composition of the Committee, responsible for the formulation of this standard is given at Annex F.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of *significant* places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

2,6 DIBROMO-4-NITROANILINE — SPECIFICATION

(First Revision)

1 SCOPE

This standard prescribes the requirements and methods of sampling and test for 2,6 dibromo-4-nitroaniline.

2 REFERENCES

The following Indian Standards contain provisions which, through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given below:

IS No.	*Title*
1070 : 1992	Reagent grade water (*third revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of dry dull yellow powder.

3.2 The material shall also comply with the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be packed in steel drums (*see* IS 2552) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

4.2 Each container shall bear legibly and indelibly the following information:

a) Name of the material;
b) Indication of the source of manufacture;
c) Net mass of material;
d) Month and year of the manufacture; and
e) Batch or lot number.

4.2.1 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 Representative samples of the material shall be drawn as prescribed in **4** of IS 5299.

5.2 Numbers of Tests

Table 1 Requirements for 2, 6 Dibromo-4-Nitroaniline

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.1)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to (4)
i)	Assay by HPLC, percent by mass, *Min*	96.0	Annex A
ii)	2-Bromo-4-nitroaniline content by GC, percent by and mass, *Max*	3.0	Annex B
iii)	4-Nitroaniline content, percent by mass, *Max*	0.2	Annex B
iv)	2,6 Dichloro 4-nitro aniline, percent by mass, *Max*	0.01	Annex C
v)	Sum of chlorobenzenes and chlorotoluenes, ppm, *Max*	50.0	Annex D
vi)	Penta chloro phenol, ppm, *Max*	50.0	Annex E
vii)	Matter insoluble in methanol, percent by mass, *Max*	0.2	**11** of IS 5299
viii)	Moisture content, percent by mass, *Max*	0.5	**10** of IS 5299

5.2.1 Tests for the determination of 2, 6 dibromo-4-nitroaniline, 2-bromo-4-nitroaniline and 4-nitroaniline shall be conducted on each of the individual samples.

5.2.2 Tests for the determination of remaining characteristics, namely, description, moisture content and volubility shall be conducted on the composite sample.

5.3 Criteria for Conformity

The lot shall be declared as conforming to, the requirement of 2,6 Dibromo-4-nitroaniline, 2-Bromo-4-nitroaniline and 4-Nitroaniline, if each of the individual test results satisfies the relevant requirements given in Table 1.

5.3.1 *For Individual Samples*

5.3.2 *For Composite Sample*

For declaring the conformity of the lot to the requirements of all other characteristics (*see* **5.2.2**) tested on the composite sample, the test “results for each of the characteristics shall satisfy the relevant requirements given in **3.1** and Table 1.

6 TEST METHODS

6.1 Tests shall be carried out according to the methods referred in col 4 of Table 1.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *SI No.* (i)]

TO DETERMINE PURITY OF 2,6 DIBROMO-4-NITROANILINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

A-1 OUTLINE OF METHOD

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

A-1.1 Objective

To determine pPurity of 2,6 Dibromo-4-Nitroanilineby high performance liquid chromatography

A-1.2 Apparatus

Binary gradient liquid chromatography system with UV detector capable of being operated under conditions suitable or resolving the individual constituents into distinct peak may be used.

A-1.3 Column

C18 100A 250 × 4.6 mm, 5µm or equivalent

A-1.4 Reagent

a) Acetonitrile — HPLC grade.
b) Water — HPLC Grade.
c) Tetra n butyl ammonium hydrogen sulphate.
d) 2,6 Dibromo-4-Nitroaniline.

A-1.5 Standard Preparation

Weigh accurately 0.050 0 g 2,6 dibromo-4-nitroaniline in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetonitrile.

A-1.6 Sample Preparation

Weigh accurately 0.050 0g m sample in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetoniltrile.

A-1.7 Buffer Preparation

Take 1.00 g tetra n butyl ammonium hydrogen sulphate in 500 ml volumetric flask. Add 200 ml HPLC grade water and complete dissolve it. Fill up to the mark with HPLC grade water.

A-1.8 Flow Rate 1.00 ml/min

A-1.9 Mobile Phase

Acetonitrile	*Buffer*
40	60

A-1.10 Column oven temperature 40^{0}C

Injection volume	5µl
Run time	25 min
Wave length	370 nm

A-2 CALCULATION

Calculate the peak area of individual constituent pertaining to 2,6 dibromo-4-nitroaniline on the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of pure to 2,6 dibromo-4-nitroaniline.

Percent of 2,6 Dibromo-4-Nitroaniline =

$$\frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

where

A_1 = area of standard2,6 Dibromo-4-Nitroaniline;
V_1 = injection volume of standard2,6 Dibrom-4-Nitroaniline;
W_1 = weight of standard 2,6 Dibromo-4-Nitroaniline;
B_1 = total volume of Standard2,6 Dibromo-4-Nitroaniline;
A_2= area of 2,6 Dibromo-4-Nitroaniline peak in sample;
V_2 = injection volume of sample;
W_2 = weight of sample; and
B_2 = total volume of sample.

ANNEX B

[*Table* 1, *Sl No.* (ii) and (iii)]

TO DETERMINATION OF 2 BROMO 4 NITROANILENE & 4NITROANILINE CONTENT BY HPLC

B-1 OBJECTIVE

To determine 2 bromo-4-nitroaniline and 4 nitro aniline in 2,6 dibromo 4 nitro aniline by high performance liquid chromatography.

B-2 APPARATUS

Binary gradient liquid chromatography system capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

B-3 COLUMN

C18 100A 250 × 4.6mm, 5µm or equivalent.

B-4 REAGENT

a) Acetonitrile — HPLC grade
b) Water — HPLC Grade
c) Tetra n butyl ammonium hydrogen sulphate
d) 2 bromo-4-Nitroaniline — Known purity
e) 4-Nitroaniline — Known purity
f) 2,6 Dichloro 4 nitro aniline — Known purity

B-5 STANDARD PREPARATION

B-5.1 Weigh accurately 0.005 0 g standard 2 bromo 4-Nitro aniline in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetonitrile — A. Take 1ml of solution A in 10 ml volumetric flask dilute it up to the mark with acetonitrile.

B-5.2 Weigh accurately 0.005 0 g standard 4-nitro aniline in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetonitrile — B. Take 1ml of solution B in 10 ml volumetric flask dilute it up to the mark with acetonitrile.

B-6 SAMPLE PREPARATION

Weigh accurately 0.050 0 g sample in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetoniltrile.

B-7 BUFFER PREPARATION

Take 1.00 g tetra n butyl ammonium hydrogen sulphate in 500 ml volumetric flask. Add 200 ml HPLC grade water and complete dissolve it. Fill up to the mark with HPLC grade water.

B-8 FLOW RATE

1.00 ml/min

B-9 MOBILE PHASE

Acetonitrile	*Buffer*
40	60

B-10 COLUMN OVEN TEMPERATURE

40^0C

B-11 INJECTION VOLUME

5µl

B-12 RUN TIME

25 min

B-13 WAVE LENGTH

370nm

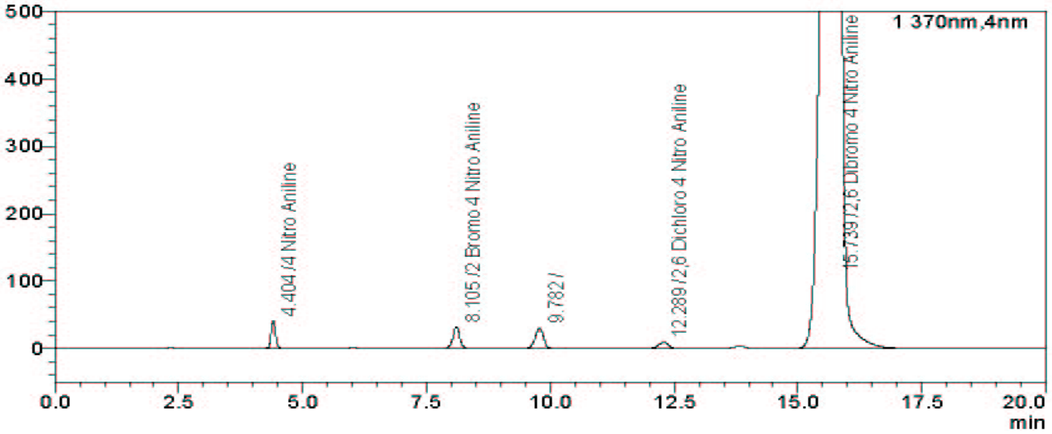


FIG. 2

B-14 PEAK TIME

4 Nitro aniline	=	4.40 min
2 Bromo-4-Nitroaniline	=	8.10 min
2,6 Dibromo-4-Nitroaniline	=	15.74 min

B-15 CALCULATION

Calculate the peak area of individual constituent pertaining to 2bromo-4-Nitroaniline/4-Nitroaniline/2,6 Dichloro-4-Nitroanilineon the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of pure to 2bromo-4-Nitroaniline/4-Nitroaniline/2,6 Dichloro-4-Nitroaniline

Percent of 2 Bromo-4-Nitroaniline =

$$\frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

Where,

A_1 = area of standard 2 bromo -4-Nitroaniline;
V_1 = injection volume of standard 2 Bromo-4-Nitroaniline;
W_1 = weight of standard 2 Bromo-4-Nitroaniline;
B_1 = total volume of standard 2 Bromo-4-Nitroaniline;
A_2 = area of 2 Bromo-4-Nitroaniline peak in sample;
V_2 = injection volume of sample;
W_2 = weight of sample; and
B_2 = total volume of sample.

ANNEX C

[*Table 1, SI* (iv)]

TO DETERMINATION OF 2,6 DICHLORO 4 NITROANILENE CONTENT BY HPLC

C-1 OBJECTIVE

To determine 2, 6 dichloro-4-nitroaniline content in 2,6 dibromo 4 nitro aniline by high performance liquid chromatography.

C-2 APPARATUS

Binary gradient liquid chromatography system capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

C-3 COLUMN

C18 100A 250 × 4.6 mm, 5µm or equivalent

C-4 REAGENT:

a) Acetonitrile – HPLC grade
b) Water – HPLC grade
c) Tetra n Butyl Ammonium Hydrogen Sulphate
d) 2,6 Dichloro 4 Nitro Aniline – Known purity

C-5 STANDARD PREPARATION

Weigh accurately 0.0025 0 g 2, 6 Dichloro 4 nitro aniline in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetoniltrile.

Take 1 ml of above solution in 100 ml volumetric flask dilute it up to the mark with acetonitrile

C-6 SAMPLE PREPARATION

Weigh accurately 0.500 0 g sample in 100 ml volumetric flask dissolve it in acetonitrile and make up to the mark with acetoniltrile.

C-7 BUFFER PREPARATION

Take 1.00 g Tetra n Butyl ammonium hydrogen sulphate in 500 ml volumetric flask. Add 200 ml HPLC grade water and complete dissolve it. Fill up to the mark with HPLC grade water.

C-8 FLOW RATE

1.00 ml/min

C-9 MOBILE PHASE

Acetonitrile	*Buffer*
40	60

C-10 COLUMN OVEN TEMPERATURE

40^0C

C-11 INJECTION VOLUME

5µl

C-12 RUN TIME

25 min

C-13 WAVE LENGTH

370 nm

C-14 PEAK TIME

2,6 Dibromo-4-Nitroaniline	— 12.29 min
2,6 Dichloro-4-Nitroaniline	— 15.74 min

C-15 CALCULATION

Calculate the peak area of individual constituent pertaining to 2,6 Dichloro-4-Nitroanilineon the chromatogram of the material. The concentration of the constituent may be obtained on the basis peak area on chromatogram obtained with known amount of pure to 2,6 Dichloro-4-Nitroaniline

Percent of 2, 6 dichloro-4-Nitroaniline =

$$\frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100$$

where

A_1 = area of standard 2,6 Dichloro-4-Nitroaniline;

V_1 = injection volume of standard 2,6 Dichloro-4-Nitroaniline;

W_1 = weight of standard 2,6 Dichloro-4-Nitroaniline;

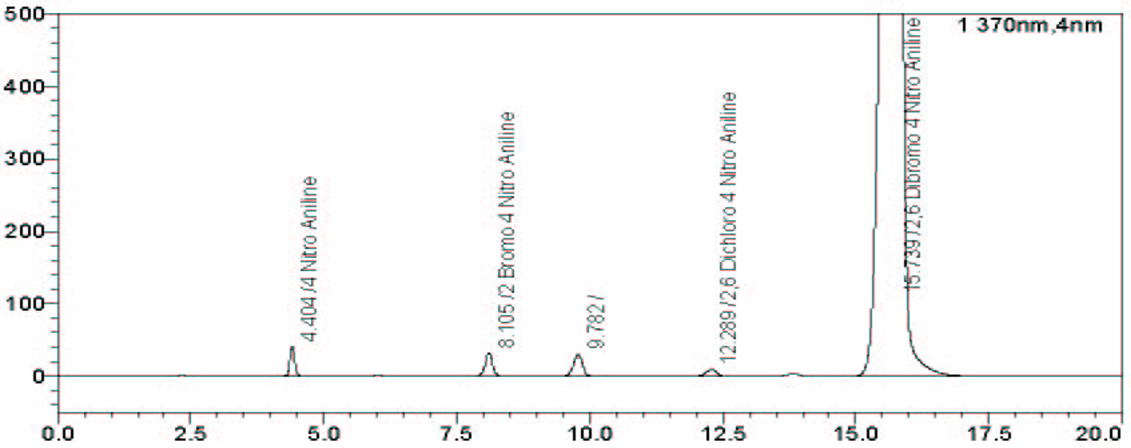
B_1 = total volume of standard 2,6 Dichloro-4-Nitroaniline;

A_2 = area of 2,6 Dichloro-4-Nitroaniline peak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample.



ANNEX D

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF CHLORO BENZENE’S & CHLORO TOLUENE’S BY GAS CHROMATOGRAPHY MASS SPECTROMETER

D-1 OBJECTIVE

To determine chlorobenzenes and chlorotouenes by gas chromatography with mass spectrometer.

D-2 APPARATUS

Gas chromatogram mass spectrometer with auto sampler.

D-3 COLUMN INFORMATION

D-4 CARRIER GAS

Helium

D-5 INJECTION PORT INFORMATION

D-6 COLUMN OVEN INFORMATION

D-7 COLUMN OVEN TEMPERATURE PROGRAM

D-8 DETECTOR INFORMATION

D-9 AUTO INJECTION INFORMATION

D-10 RETENTION TIME OF THE CB’S

Name	*Film Thickness*	*Column Length*	*Inner Diameter*	*Column Max. Temp.*
ZB-35 or equivalent	0.25 um	30 m	0.25 mm ID	340 ^{0}C

Injection Mode	*Temp.*	*Column Flow*	*Purge Flow*	*Split Ratio*	*Injection Volume*
Split	240 ^{0}C	1.00 ml/min	3.0 ml/min	2.0	1micro liter

Initial Temp.	*Equilibration time*	*Total Program Time*
40 ^{0}C	3.00	36 min

Rate (^{o}C/min)	*Temperature (^{o}C)*	*Hold Time (Min)*
–	40	0.5
5	140	1
20	290	6

Detector	*Iron Source (EI) Temp.*	*Interface Temp.*	*Solvent Cut Time*	*Detector Voltage*	*Threshold*
MS	230 ^{o}C	280 ^{0}C	3 min	Relative + 0.3 kV	100

Start Time	*End Time*	*Acquire Mode*	*M/Z*
3	11	SIM	63,77,91,112,114,126
11	15	SIM	65,91,111,114,146,148
15	20	SIM	125,127,145,160,180,182
20	26.5	SIM	159,161,194,196,214,218,240,250,252
26.5	29.5	SIM	229,284,286

Rinses with Solvent (pre Run)	*Rinses with Solvent (post Run)*	*Rinses with Sample*	*Plunger speed*	*Viscosity Comp. Temp*	*Plunger Speed*	*Syringe Inserting Speed*
0	5	2	High	0.2 sec	High	High

Product Name	*R. Time*	*M/Z*		*Product Name*	*R. Time*	*M/Z*
MCB	6.59	112		Toluene	4.43	91
MDCB	11.33	146		2-CT	9.52	91
PDCB	11.68	146		3-CT	9.59	91
ODCB	12.57	146		4-CT	9.65	91
1,3,5 TCB	14.97	180		Alpha CT	12.36	91
1,2,4 TCB	16.83	180		2,4 DCT	14.67	125
1,2,3 TCB	18.21	180		2,6 DCT	14.96	125
1,2,3,5 TeCB	21.04	216		2,5 DCT	14.91	125
1,2,4,5 TeCB	21.13	216		3,4 DCT	15.87	125
1,2,3,4 TeCB	22.88	216		2,3 DCT	15.97	125
Penta CB	24.93	250		2,4,5 TCT	20.04	159
Hexa CB	26.75	284		Penta CT	26.44	229

D-11 REAGENTS

a) mono chlorobenzene [MCB]
b) 1,2-dichlorobenzene [ODCB]
c) 1,3-dichlorobenzene [MDCB]
d) 1,4-dichl orobenzene [PDCB]
e) 1,2,3-trichlorobenzene [123 TCB]
f) 1,2,4-trichlorobenzene [124 TCB]
g) 1,3,5-trichlorobenzene [135 TCB]
h) 1,2,3,4-tetrachlorobenzene [1234 TeCB]
j) 1,2,3,5-tetrachlorobenzene [1235 TeCB]
k) 1,2,4,5-tetrachlorobenzene [1245 TeCB]
m) penta chlorobenzene [PCB]
n) hexa chlorobenzene [HCB]
p) toluene
q) 2-chlorotoluene [2 CT]
r) 3-chlorotoluene [3 CT]
s) 4-chlorotoluene [4 CT]
t) 2,3-dichlorotoluene [23 DCT]
u) 2,4-dichlorotoluene [24 DCT]
v) 2,5-dichlorotoluene [25 DCT]
w) 2,6-dichlorotoluene [26 DCT]
y) 3,4-dichlorotoluene [34 DCT]
z) 2,3,6-trichlorotoluene [236 TCT]
aa) 2,4,5-trichlorotoluene [245 TCT]
bb) penta chlorotoluene [PCT]
cc) Di chloro methane [DCM]
dd) Methanol

D-12 STANDARD PREPARATION

a)	Weigh 0.002 5 g each standard in each 25 ml volumetric flask and fill to the mark with methanol — Stock solution of each standard. — 100 ppm each solution.	Stock solution
b)	Take 1ml of each stock solution in 100 ml volumetric flask and fill the mark with DCM. [1 ppm each mix. solution]	Stock solution No.1., except 2,6 DCT and 3,4 DCT.
c)	Take 10 ml of above stock solution No.1 in 100 ml volumetric flask and fill the mark with DCM. [0.1 ppm each mix. solution].	Stock solution No. 2.
d)	Take 5 ml of above stock solution No.1 in 100 ml volumetric flask and fill the mark with DCM. [0.05 ppm each mix. Solution].	Stock solution No. 3.

e)	Take 1 ml of above stock solution No.1 in 100 ml volumetric flask and fill the mark with DCM. [0.01 ppm each mix. Solution].	Stock solution No. 4.
f)	Take 10 ml of above stock solution No.3 in 100 ml volumetric flask and fill the mark with DCM. [0.01 ppm each mix. Solution].	Stock solution No. 5.

D-13 SAMPLE PREPARATION

Weigh 0.1 g sample into a 10 ml volumetric flask. Add 5 ml DCM and shake well on cyclo mixer. After 30 min fill to the mark with DCM. The solution has to be filtered before injection into the GC-MS.

If necessary dilute the sample solution according the calibration range.

D-14 CALCULATION

$$\text{CBs/CTs, in ppm} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100 \times 10\,000$$

where

A_1 = Area of standard CBs / CTs;
V_1 = Injection volume of standard CBs/CTs;
W_1 = Weight of standard CBs / CTs;
B_1 = Total volume of standard CBs / CTs;
A_2 = Area of CBs / CTs peak in sample;
V_2 = Injection volume of sample;
W_2 = Weight of sample; and
B_2 = Total volume of sample.

ANNEX E

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF PENTA CHLORO PHENOL (PCP) BY GAS CHROMATOGRAPHY MASS SPECTROMETER

E-1 OBJECTIVE

Determination of Penta Chloro Phenol (PCP) in 2,6 Dibromo 4 Nitro Aniline by gas chromatography mass spectrometer.

E-2 EQUIPMENT

E- 2.1 Gas chromatograph mass spectrometer with auto sampler.

E-2.2 Column — DB-35-MS 30 m * 0.25mm, film thickness * 0.25 µm or equvivelent

E-3 GC PARAMETER

E- 3.1 Injector

Injection Mode	*Temperature*	*Split Ratio*
Split	240 ºC	5

E-3.2 Carrier Gas — Helium

E-3.3 Injection Volume — 1 µl

E-3.4 Oven Temperature

Heating Rate (°C/min)	*Temperature (°C)*	*Hold Time (min)*
--	90	0.00
5	130	0.00
2.5	145	0.00
7	205	0.00
25	300	9.63

3.5 MS PARAMETER

Note — Aging of the column can influence the sepration so that the conditions, parameters like flow may have to changed.

Ion Source Temperature (°C)	*Interface Temperature (°C)*	*Solvent Cut off*	*Detector Voltage*	*Threshold*
230	280	3.5	Relative to the Tuning +0.2 kV	400

Start Time	*End Time*	*Acquire Mode*	*M/Z*
22.5	24.5	SIM	248, 260, 262, 264, 266, 308, 332

E-4 REAGENTS AND APPARATUS

a) 0.1M Potasium carbonate solution in water;
b) Methanol AR grade;
c) Acetic anhydride;
d) Triethylamine;
e) Hexane;
f) Pentachlorophenol;
g) Sodium sulphate;
h) Parrafilm;
j) Rotary flask shaker; and
k) Ultra sonic bath.

E-5 PREPARATION OF STANDARD

a)	Weigh 0.002 5 g each standard in 25 ml volumetric flask and fill up to the mark with methanol — Stock solution of each 100 ppm	Stock solution
b)	Take 1 ml of each standard stock solution in 100 ml volumetric flask and fill the mark with Methanol [1ppm each mix solution].	Stock solution No. 1
c)	Take 10 ml of above stock solution No.1 in 100 ml volumetric flask and fill the mark with methanol [0.1ppm each mix solution].	Stock solution No. 2
d)	Take 50 ml of above stock solution No.1 in 100 ml volumetric flask and fill the mark with methanol [0.5 ppm each mix solution]	Stock solution No. 3
e)	Take 2 ml of above stock solution No.1 in 100 ml volumetric flask and fill the mark with methanol [0.02 ppm each mix solution]	Stock solution No. 4

Take 10 ml of 0.1 M potassium carbonate in 25 ml volumetric flask, add 0.5 ml standard solution. Then add 4 ml acetic anhydride and 0.5 ml triethyl amine and close the volumetric flask with paraffin tape with minor hole to release carbonate gas. Then shaking the flask for 1 h on rotary flask shaker. After the acetylating reaction complete at room temperature, add 5 ml n-Hexane then put the flask on rotary flask shaker for shaking 1 h. Then after transfer the solution in test tube and place the test tube for 15 min in ultrasonic bath for layer separation of n-Hexane. Then filter the layer of n-Hexane through whatman filter paper no. 42 with use 1 g sodium sulphate for dry the n-Hexane phase. Use the dry n-Hexane filtrate for GCMS analysis.

E-6 SAMPLE PREPARATION

Weigh 0.1 gm of the sample in to 25 ml volumetric flask add 10 ml of 0.1 M potassium carbonate. Then add 4 ml acetic anhydride and 0.5 ml triethyl amine and close the volumetric flask with paraffin tape with minor hole to release carbonate gas. Then shaking the flask for 1 h on rotary flask shaker. After the acetylating reaction complete at room temperature add 5 ml n-Hexane then put the flask on rotary flask shaker for shaking 1 h. Then after transfer the solution in test tube and place the test tube for 15 min in ultrasonic bath for layer separation of n-Hexane. Then filter the layer of n-Hexane through whatman filter paper no. 42 with use 1 g sodium sulphate for dry the n-Hexane phase. Use the dry n-Hexane filtrate for GCMS analysis.

E-7 RETENTION TIME

Retention Time	*Base ion M/Z*	*Reference ions M/Z*	*Component*
23.88	266	264,304	pentachlorophenol

E-8 CALCULATION

$$\text{PCP in ppm} = \frac{A_2 \times V_1 \times W_1 \times B_2}{A_1 \times V_2 \times W_2 \times B_1} \times 100 \times 10\,000$$

where

A_1 = Area of STD PCP;
V_1 = Injection volume of STD PCP;
W_1 = Weight of STD PCP;
B_1 = Total volume of STD PCP;
A_2 = Area of PCP peak in sample;
V_2 = Injection Volume of sample;
W_2 = Weight of sample; and
B_2 = Total volume of sample.

ANNEX F

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
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Alkyl Amines Chemicals Limited, Mumbai	Shri Kirat Patel Shri S. V. Nikumbhe (*Alternate*)
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BASF India Limited, Mumbai	Shri Uday Kulkarni
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Colourtex Industries Limited, Mumbai	Dr Pankaj Desai Shri Ismail Habibulla (*Alternate*)
Deepak Nitrite Limited, Vadodara	Shri Sailash Raval Dr Mangalya Kar (*Alternate* I) Dr J. K Astik (*Alternate* II)
Ecological and Toxicological Association of Dyes (ETAD), Vadodra	Dr Pariti Siva Rama Kumar
Gujarat Dyestuffs Manufacturers Association (GDMA), Ahmedabad	Shri R. S. Patel Shri Bansibhai Patal (*Alternate*)
Gujarat Pollution Control Board (GPCB), Ghandhinagar, Ahmedabad	Shri Y. A. Tai
Heubach Colour Private Limited, Mumbai	Shri J. I. Sevak Shri Vinod Madhukar (*Alternate*)
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The Bombay Textile Research Association	Dr Ashok N. Desai
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